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PROJECT 325B

MONTHLY SUMMARY REPORT

PERIOD: April 1, 1972 to April 30, 1972

Submitted By: [redacted]

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Project Manager

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PROJECT 325B

SUMMARY REPORT

PERIOD: April 1, 1972 to April 30, 1972

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SUBMITTED BY: [redacted]

Project Manager

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SUMMARY

Project work has continued at a high level during the month of April and in most task areas follows the original plan of work and time schedules.

CHEMICAL R and D

Improvements continue to be made in yields of photograde D260.

D259, a precursor of D260, has been positively identified as an impurity in D260.

Purifications and preliminary film evaluations have been completed for a series of Leuco Malachite Green (LMG) analogs. In all cases the photosensitivities are vastly inferior to D260 and LCV.

An apparently pronounced synergism between D260 and CBr_4 has been identified. As a result of improved D260 purity previously labeled "bad" CBr_4 is now found to give very good 5/D7 films.

Binder purification studies have continued, but have been delayed due to mechanical difficulties.

Work has begun to study the effects of polystyrene molecular weight.

A purified air chamber has now been used successfully to produce good quality films.

A color interaction has been observed between benzene and D7. The purity of benzene is now suspect and is being investigated.

Work on the 5/DPA was begun. Preliminary results appear superior to those last obtained in late 1970. This system has been found to markedly differentiate samples of D260 which give comparable and hence indistinguishable films with 5/D7.

Preliminary work began on the Dye Identification program. This work was hindered as a result of two man weeks lost due to illnesses.

1.0 CHEMICAL R AND D

1.1 Leuco Dye Program

1.1.1 D260

A dramatic improvement in yield of photograde D260 has now been realized by using dry column chromatography. The yield has been raised to 84% from the 48-61% yields realized by the trituration and recrystallization procedures described last month.

Some one dozen small scale purification procedures were also investigated this month. Details of these studies will not be elaborated, but it is noted that differently prepared samples of D260 are found to give essentially indistinguishable results with 5/D7 but give quite dramatic differences with 5/DPA (see Section 1.6.1). These different samples are indistinguishable by TLC except that small and different amounts of D259 and anthrone are present as impurities. Addition of D295 and/or anthrone to either sample failed to alter its behavior significantly. It is therefore concluded that the differences noted by the DPA system must be due to an undetected impurity. Mass spectral studies as well as repeats of the purification procedures are underway to detect the impurity(s). It is suspected that the impurity may be D260-hydroperoxide or similarly related peroxides. Since the hydroperoxide and other related peaks have been seen in mass spectra of D260 samples, it is hoped that direct and careful comparison of these different D260 samples will aid in clarifying this suspicion.

1.1.1.1 D260 Impurity Studies

Work continues to identify gross and trace impurities in crude and purified D260. Although D260 does not fluoresce under long UV light, it was discovered that D259 does fluoresce and is present as a significant impurity in crude D260. Virtually all D259 is removed from crude D260 by ethyl ether trituration or by column chromatographic purification.

Decomposition of hydroperoxide of D260 has been postulated as one source of anthrone and concurrently, 4DMAP. Prolonged irradiation of a benzene solution of D260 with an intense UV source rapidly increased the amount of anthrone present.

Irradiation in excess or in small amounts of air did not qualitatively change the rates of decomposition of two different samples. Since anthrone is detected by UV on TLC at a much lower level (10 ppb) than 4DMAP is detected by visual spray (0.01%), irradiation was continued for a prolonged period. At the end of 10 to 12 hours, a spot corresponding in Rf value and color to 4DMAP was detected in this irradiated sample. Since the presence of microgram quantities of 4DMAP in the film system has been shown to be deleterious, the visualization by TLC of even 10 ppb of anthrone may signal that enough 4DMAP is concurrently present to be damaging. Anthrone itself has been shown not to be damaging up to the milligram level.

Work continues in an attempt to demonstrate a one-to-one relationship of anthrone to 4DMAP. It will be remembered that such a relationship provides convincing evidence for the presence of D260 -hydroperoxide.

Purified D260 (2 g) was stirred with $NaBH_4$ in methanol in an attempt to eliminate anthrone. A TLC indicated anthrone was still present.

1.1.1.2 Anthrone Route to D260 and Analogs

1.1.1.2.1 Carbinol Resynthesis

In a repeat of the first oxidation in March, another 10 g of D263 was oxidized to carbinol by the Aaron and Barker procedure, using lead dioxide paste. The yield was doubled, now closely approximating the literature report.

1.1.1.2.2 Anthrone Synthesis and Purification

The carbinol formed above was dismuted according to Aaron and Barker to give 6.45 g of crude anthrone. Work throughout the month on the column purification of the anthrone hammered home the true meaning of "dismutation" as used by Aaron and Barker. Carbinol does disproportionate between D263 and anthrone. The many samples that were being chromatographed this month all contained D263, carbinol and anthrone. Highly colored impurities are left behind by column chromatography, but a good separation of anthrone from carbinol may require chromatography with automatic sample collection on a tall, thin column. D263 is the most mobile

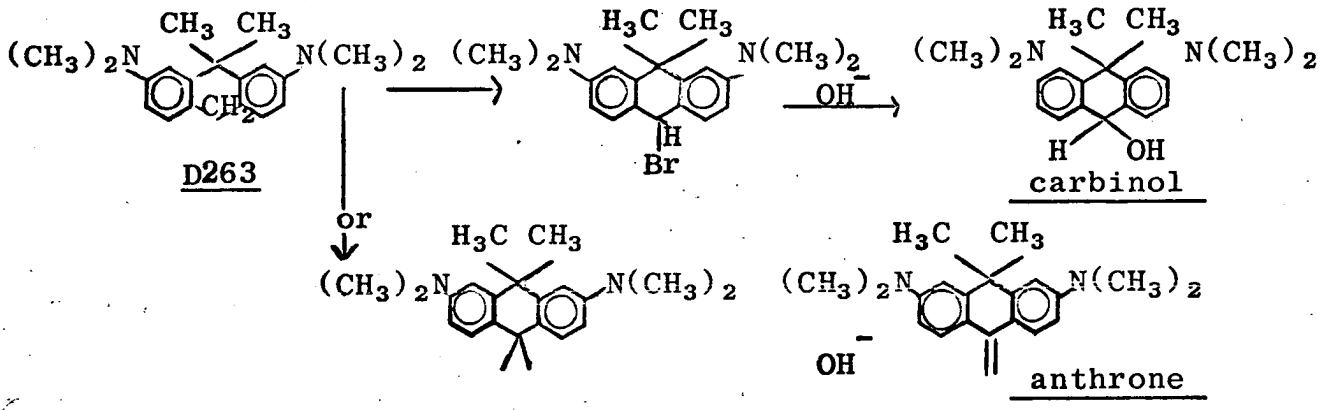
component, fairly well separated from anthrone and carbinol. The latter two move quite slower and are difficult to separate.

Recrystallized anthrone has been obtained, mp 210-211° (Aaron and Barker mp 207-208°), as pale chalky-green crystals.

This procedure has been abandoned, at least temporarily, to provide time to explore more attractive possibilities to the anthrone.

1.1.1.2.3 Attempted Brominations of D263

Two attempts were made to brominate D263 with N-bromo-succinimide. The objective was to form mono- or dibrominated derivatives of D263 which should yield carbinol or anthrone, respectively:



Visual indications of a reaction was immediately evident. There was a rapid color change from a pale green blue to a bronze-red solid on the walls of the flask, although the solution phase was not highly colored. TLC's of both runs showed only very faint spots which might have been brominated products. The reaction mixtures on TLC observation showed anthrone, D263 and other products.

Bromination of D263 with bromine gave equally complex and unsatisfactory results. D263 was reacted with equivalent amounts of bromine and of triethylamine as an acid scavenger at 25° for one hour and 45° for almost three hours. Since the TLC showed a very high concentration of D263 remaining unreacted, additional equivalents of bromine and of triethylamine were added, the charge thoroughly stirred and allowed to stand over night at room temperature. The following day the reaction was heated at 80° for an additional three hours.

Both bromination methods led to complex mixtures containing very large concentrations of unreacted D263, along with some anthrone and some probably brominated products. Neither route seemed promising enough for further qualitative investigation at this time.

1.1.1.2.4 Attempted Oxidations of Carbinol

Small qualitative oxidations of impure carbinol were carried out in DMSO under oxygen. Although no more anthrone was found than in the crude starting material, the amount of carbinol increased at the expense of the D263 and to a greater extent as the reaction temperature rose from 25 to 160°. This may be a possible alternate oxidation of D263 to carbinol at the higher temperatures.

1.1.1.3 Film Studies

1.1.1.3.1 Aging of Coating Solutions for Formula 5 and 5/D7

A standard Formula 5 coating solution (D260, 4PO, CBr_4) was made and stored in the dark at ambient temperature. Aliquot portions were taken at measured times, coated and developed (optical and printout mode). Table I contains the results.

Optical Development - Because of the high blue fog background (fog density .40) and low maximum density (.64) producted in a freshly coated film, one cannot fully determine the effect of aging of the coatihg solution used to make that film, but the following trends can be seen:

1. As the solution ages, the development time needed to produce a nearly constant maximum density of 0.60 to 0.70 decreases.

2. After aging 30 minutes, the Dnet decreases to a point (0.10) where it becomes difficult to differentiate between image and fog. After 2-1/2 hours, one can barely make out the image in the fog background while after 19 hours, no image at all can be seen in the fogged film.

An earlier study (February Summary Report) showed that the normal 5/D7 coating solution exhibited significant decay

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after 2 to 3 hours. The results of that study are shown again in Table 2.

To determine the effect of D7, it was added to the Formula 5 solution just prior to coating. The results are seen in Table 3. First, the image enhancing property of D7 is immediately evident by comparing Tables 3 and 1 but the decay trends are parallel. Comparison of Table 3 with 2 shows a significant differences in rate of density and development time decays. The comparison indicates that the presence of D7 during aging results in better films, suggesting perhaps some retardation of decay. If so this contrasts the results of the ingredient interaction studies reported last month where in the absence of CBr_4, D7 showed deterimental interaction with D260. Perhaps the most significant differences is the decrease in development time as opposed to the dramatic increase exhibited by the interaction studies where CBr_4 was not present during decay.

The inescapable conclusion from all the solution decay studies is that all significant interactions are observed only when D260 is present during the decay. This is readily evident from the 5/D7 solution without D260 which showed no similar decay after 52 days (see Table 4).

1.1.2 Other Leuco Dyes

Work finally proceeded this month in purifying the Leuco Malachite Green (LMG) analogs most of which were synthesized late last year. Nearly all of the crude materials showed numerous impurities by TLC. Each was purified by recrystallization until TLC showed a single spot material. To gain some ideas of the possible effects of impurities, film evaluations were made on both crude and purified materials in most instances. The results of these evaluations are seen in Table 5; the crude materials are denoted by "C" following the Dye Index number.

The evaluations were made by comparison to Leuco Crystal Violet (LCV) in relation to both Formula 5 which contains 100 mg of D260, and Formula 7 which contains 150 mg of LCV. The comparison to Formula 5 was made on the basis of molar equivalent to D260 while the comparison to Formula 7 (LCV) was made on a weight equivalent basis (150 mg). The structures of the leuco dyes are shown in Table 6. Where possible a preliminary speed decay evaluation was made by repeating the printout exposure after three (3) hours. These results are also shown.

By comparison to LCV (nos. 1 - 4) it is apparent that all the other leuco dyes are vastly inferior. Only D411 (no. 5) approaches LCV and it is in fact an analog of LCV rather than LMG.

Comparison of crude and purified samples shows no dramatic differences with the exception of color differences in some instances (e.g. 10, 11, 12, and 13).

Rapid speed decay is also evident in every case tested.

The results of these studies thus far, suggest that removal of the dimethylamino group from LCV to give LMG and its various analogs is responsible for the much lower densities. In several instances the sensitivities may not be significantly less if the number of steps produced is accepted as a fair basis for comparison. Thus, D413 (no. 13) and D405 (no. 16) both show seven (7) steps in comparison to eight (8) steps for LCV.

Care must, however, be exercised in concluding that the poorer properties of LMG and its analogs are due to absence of the third dimethylamino group. The severe retarding effects which can be exhibited by traces of impurities has already been amply illustrated in the case of D260. There is no guarantee that trace impurities are not responsible here. One way to assure that impurities are not responsible is to repeatedly purify and evaluate each leuco compound. If photoproperties remain constant or approach a constant behavior it would seem probable that the structure and not impurities is responsible. On the other hand the presence of new groups such as methyl, bromo and nitro might be responsible if it were not for the fact that LMG itself is apparently no better than its analogs, at least so far as density is concerned.

In order to gain further insight into the question of structure versus purity, several LCV analogs, the 2-methyl and 2,6-dimethyl derivatives, are being synthesized. These two leucos have the third dimethylamino group and additional methyl groups for comparison to methylated LMG analogs (D405, D406, and D409). If the behavior of these derivatives are more like LCV than it would appear safe to conclude that the absence of the dimethylamino group is the major cause of the poorer photoproperties of LMG and its analogs.

1.2 CBr_4

It has been discovered that so called "bad" samples of CBr_4 in fact give excellent 5/D7 films with D260 of recent

preparation and purification. It appears that a pronounced synergism exists between D260 and CBr_4 similar to the one between 4PO and CBr_4 which was recognized last year. Further clarification of this discovery is not possible at this time, but once uniform and optimum purities of all materials are realized it should be possible to verify this observation. Of immediate interest of course is the fact that the CBr_4 purity problem and the so called solid state deterioration may not be the problem as has been thought to be.

The solid state deterioration study continues. Two evaluations were made during April and all samples remain unchanged. Of course this may now be due to the fact that higher quality D260 is being used.

1.3 N-Oxide Program

Indefinitely postponed--nothing to report.

1.4 Binder Studies

1.4.1 Purification and Deoxygenation

As a prelude to further ingredient interaction decay studies three polystyrenes were specially treated to remove oxygen, residual water and other volatile contaminants. Samples of Styron 685, 686 and 200K from Pressure Chemical were charged to 2 liter round bottom flasks attached to vacuum manifold. Alternate purging with argon and evacuation while heating at 225-240°C was carried out for several days. All three of these flasks eventually cracked. It was then thought that heavy walled resin kettles would be more suitable . After two and one-half days heating, one sample, the Pressure Chemical material, was completely degassed and the sample was transferred under high vacuum (0.05 mm) to the oxygen-free environmental chamber. The other two flasks, not completely degassed, were cooled for the weekend to be left under high vacuum. On cooling to room temperature the one containing Styron 686 suddenly cracked. This is attributed to the polystyrene's capacity to bond firmly to the glass to produce stress as the two materials then cool with great differences in coefficients of expansion. To alleviate this problem, Teflon-lined resin kettles have been ordered. The final flask containing the 685 has been held under high vacuum and an attempt will be made to complete the deoxygenation.

These treated polymers will be used to study effects of polymer along with oxygen on the various ingredient interactions (and hence on the overall speed decay problem) which were described last month (Section 1.1.1.4).

The polymers after degassing are being transferred to the Vacuum Atmosphere's oxygen-free chamber for dissolution in deoxygenated benzene. The equipment difficulties described in the preceding paragraph have resulted in a further delay of this study. Furthermore, the fused nature of the treated polystyrenes results in long dissolution times, on the order of nearly a week. This fact will further delay completion of the overall study. It currently appears that it will be the end of May before the entire study is underway and it will therefore be the end of June before all the results are completed.

1.4.2 Effects of Molecular Weight

Preliminary studies were initiated to study the effect of polystyrene's molecular weight on sensitometric and speed decay properties. Pressure Chemical's various molecular weight standards are being used and comparisons made to the 200K which is essentially identical in behavior to MX4500 (cf. March Summary Report, Section 1.4.1, p. 10).

In order to properly evaluate the effects of molecular weight, constant film thickness and chem/binder ratio must be maintained since both have pronounced effects on sensitometric properties.

Molecular weight ranging from 20K to 2 million (2M) are planned for study. As a result, the standard 10% solutions vary in viscosity from water thin to near immobility (see Table 7). In order to obtain good quality coatings the viscosities must be altered which in turn alters chem/binder ratio. Once a suitable coating viscosity is obtained, however, a constant chem/binder ratio and dry thickness can be attained by appropriately varying the wet coating thickness.

The results to date are shown in Table 7. They show that although viscosities vary considerably, the resulting dry thicknesses do not vary significantly. This will greatly facilitate the study since a wider range of viscosities can be used. Work proceeds in order to standardize the procedure for each molecular weight involved. Once this is accomplished the study will be performed under a controlled atmosphere (see Section 1.5.1), with particular attention to the effects on speed decay.

1.5 Environmental Studies

1.5.1 Controlled Air Environment

A large dry box has been received from Kewaunee Scientific for use in our controlled air environment experiments. The box is of sufficient size to permit formulation, coating and exposure of films. The box has four glove ports, two on each side, which permits two men to work in the box if necessary. The box has been purged with synthetic air (80% N_2, 20% O_2) and is equipped with a circulation system with purification train.

The box has been fitted with balance, exposure unit and all other necessary formulation and coating equipment. Several weeks effort has been spent in coating 5/D7 to establish proper methods and procedures. Considerable difficulty was encountered with static both in weighing materials and in coating uniform films. These difficulties have now been overcome and good quality 5/D7 films have been obtained. The following procedures were found necessary:

1) A polonium α-source on the balance.

2) Interior of box washed periodically with an antistatic solution.

3) Circulator pump off during coating.

4) Maintain slight positive pressure in box.

1.5.2 Materials' Purifications

Now that the chamber is in operating order work was begun on final purifications of materials. The plan is to recrystallize D260, D7 and 4PO from pure benzene under a pure air or argon environment. By using pure benzene, the same which is used for coating, possible contamination from other solvents is avoided. By recrystallizing under pure air or inert environment the possibility of environmental contamination is avoided. In this way we hope to demonstrate whether or not air borne contamination, which has been encountered during film evaluation, is also indirectly contributing to less than optimum results through contamination of the individual ingredients prior to formulating.

Work began by recrystallizing D7, which to date has been recrystallized from pyridine/petroleum ether. D7 is a brilliant yellow compound which forms bright yellow solution in benzene and other solvents. In attempting to recrystallize D7 from benzene, we have noted that upon heating to about 40°C, the yellow solution turns orange and the subsequently recrystallized D7 is orange, not yellow. We then used benzene which was washed with concentrated sulphuric acid, followed by caustic wash, water wash and drying. In this instance a D7 solution turned only slightly orange at reflux (80°C). A subsequent, larger scale batch of benzene afforded a color change at 55°C.

From these observations it appears that an acid-base reaction is occurring to produce a small quantity of D7-dye. The washed benzene may contain enough residual caustic to affect this equilibrium, thus explaining the differences in temperatures at which the color change was noted. Another explanation involves the fact that even reagent benzene can contain traces of higher (naphthenic) aromatics. These may be reacting with D7 to produce new dyes or perhaps pi-complexes. One would expect that washing with concentrated sulphuric acid would remove many of these higher aromatics in the same way that thiophene is removed. We are currently awaiting arrival of a spinning band column in order to complete a scrupulous purification of benzene. It is also noted that the color changes due to acid contamination could be resulting from atmospheric contamination since we have not avoided this possibility to date.

With this discovery it is apparent that the purity of benzene is now a potential suspect in the question of shelf life/speed decay. The questions concerning its purity as evidenced by the D7 interaction is being actively investigated. Obviously there is no point to running carefully controlled experiments and purifying key ingredients using benzene whose integrity is suspect. Until the benzene issue is resolved the controlled environmental coating experiments will be delayed. On the basis of our observations to date it appears that carefully washing benzene with sulfuric acid may be sufficient to avoid the color change with D7. If this is so then film evaluations will be made comparing the treated benzene and the regular benzene and the purification of materials can proceed using the treated benzene.

1.6 Other Film Systems

1.6.1 Formula 5/DPA

Work began this month to study the 5/DPA system. Our initial objective is to duplicate those promising film results which were

realized in late 1970. It will be remembered that with the advent of materials' problems in early 1971 the 5/DPA also failed. We were subsequently unable to reinstate the system in contrast to 5/D7. It was then postulated that the principle reason for this was D260 and that 5/DPA was more sensitive to its disposition than was 5/D7.

Our studies therefore began with investigating various samples of D260 (cf. Section 1.1.1). Evaluations were made using a 15 minute printout exposure ($E = 1.5 \times 10^5$ mcs) which is comparable to that obtained in October 1970 (Figure 1-A). The first D260 to be evaluated was that which has been more or less the standard in recent months. It is crude D260 which has been recrystallized from cyclohexane (15R). A 15 minute printout afforded a high fog film: Figure 1-C.

Crude D260 was then recrystallized from benzene/petroleum ether to afford three crops. The first crop afforded results similar to curve 1-C. Unfortunately the second crop was blended with the first crop to provide sufficient material to maintain the 5/D7 studies. This was done prior to the beginning of the 5/DPA investigations and so the second crop was not available for testing. The blended sample (CS-3W), however, gave in marked contrast curve B (Figure 1). This curve is in fact better than was obtained in 1970. It also was developable to give curve B in Figure 2. By contrast, crop 1 as well as sample 15R totally fogged with little or no discernible image. Crop 3 which was only a very small amount also gave a printout similar to 1-B though slightly less sensitive. There was insufficient material to evaluate in the development mode. By contrast to the markedly different behaviors in 5/DPA both CS-3W and 15R gave comparable 5/D7 curves as seen in Figure 3. The differences in curve shape and density may be a manifestation of the differences seen in 5/DPA, but this is uncertain due to the current range of error associated with 5/D7.

TLC comparisons of D260 showed varying amounts of D260-anthrone and D259 as impurities but cross experiments (see Section 1.1.1) ruled these out as causes for the sensitometric differences. Thus, a currently undetected impurity appears to be the only plausible explanation for these differences. Samples of 15R, CS-3W and the third crop have been sent for mass spectral analysis. It is hoped this will identify the impurity(s) responsible and particular note will be made of hydroperoxide and peroxide contents. A repeat of the purification procedure is planned.

This work to date is a further illustration of the complexity of the D260 purity issue and the associated photomechanisms which must be involved. The success realized thus far with 5/DPA is a further illustration of the potential gains to be realized by careful attention to materials' purities and the D260 purity in particular. As work continues the past complications and difficulties of these systems are becoming understood, and there is now every reason to be confident that excellent film reliability and repeatability will soon be realized.

1.7 Film Analysis of Decay Products

Nothing to report.

1.8 Dye Identification

Work began this month in this area. Unfortunately the effort was considerably less than planned due to several illnesses which totalled losses of two man weeks. However, preliminary studies with fixed film has resulted in TLC identification of two dyes, one blue the other red, as well as a third non-dye component. Work continues to isolate and identify these products as well as to seek still others.

1.9 System Nonuniformities

Nothing to report.

1.10 Miscellaneous Studies and Discussion

Nothing to report.

TABLE 1

AGING OF FORMULA 5 COATING SOLUTION

Exposure Number 1824-	γ	AEI	Dmax	Dmin	Dnet	Δ^{T_a}	Δ^{T_r} sec.
Optical Development (T_e = .09)							
42-6A	-	-	.64	.40	.24	2 min.	70
-7A	-	-	.52	.42	.10	30 min.	55
-8A	-	-	.46	.38	.08	1 hr.	45
-9A	-	-	.67	.60	.07	1.5 hrs.	42
-10A	-	-	.70	.65	.05	2.5 hrs.	35
43-1A	-	-	.60	.59	.01	19 hrs.	25
Printout Mode (T_e = 300 sec.)							
42-6	.9	1.6×10^{-4}	1.23	.06	1.17	2 min.	
-7	.9	2.2×10^{-4}	1.24	.05	1.19	30 min.	
-8	.9	2.2×10^{-4}	1.26	.06	1.20	1 hr.	
-9	.9	2.2×10^{-4}	1.24	.07	1.17	1.5 hrs.	
-10	.70	3.5×10^{-4}	1.32	.10	1.22	2.5 hrs.	
43-1	.70	2.2×10^{-4}	1.21	.16	1.05	19 hrs.	

TABLE 2*

AGING STUDY OF STANDARD 5/D7 COATING SOLUTION

Exp. No. 1769	γ	AEI	Dmax B	Dmin B	Dnet B	ΔT_r sec.	Curve Smooth	Curve Shape	ΔT_a
48-2	1.30	1.10	2.14	.22	1.92	120	2	2	2 min.
43-9	1.80	.40	2.06	.26	1.80	120	1	1	5 min.
43-10	2.60	.35	2.07	.28	1.79	130	1	1	10 min.
43-11	1.60	.40	1.76	.22	1.54	120	1	1	15 min.
44-2	1.80	.55	2.06	.33	1.73	120	1	1	20 min.
44-3	3.00	.50	2.44	.37	2.07	120	1	1	30 min.
44-4	1.00	1.20	1.52	.38	1.14	110	1	0	40 min.
44-5	-	-	1.70	.33	1.37	110	-	-	50 min.
44-6	1.5	.50	1.50	.33	1.17	100	0	1	1 hr.
44-7	2.40	..13	1.90	.40	1.50	90	1	1	2 hrs.
44-8	-	.1	1.42	.37	1.05	90	-	-	3 hrs.
44-10	1.30	.15	1.30	.36	.94	90	1	0	4 hrs.

ΔT_a = Time lapse after addition of activator(s) to coating solution.

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TABLE 3

AGING OF FORMULA 5 COATING SOLUTION (D7 ADDED JUST PRIOR TO COATING)

Exposure Number 1824-	γ	AEI	Dmax	Dmin	Dnet	ΔT_r sec.	ΔT_a	Comments
Optical Development (T_e = .09)								
43-2	1.90	1.12	1.90	.21	1.69	32	0	Control
-3	1.30	1.4	1.43 m	.21	1.22	35	5 min.	
-5	-	1.0 (?)	1.20	.23	.97	26	30 min.	Some Blotch
-7	-	-	1.24	.30	.94	17	2 hrs.	Blotch
-9	-	-	1.00	.33	.67	17	3.5 hrs.	Blotch
-11	-	-	.73	.53	.20	17	5.5 hrs.	Blotch
Printout Mode (T_e = 300 sec.)								
43-4	1.5	3.5×10^{-4}	2.40	.09	2.31		5 min.	
-6	1.3	3.5×10^{-4}	2.30	.10	2.20		30 min.	
-8	1.4	5×10^{-4}	2.42	.12	2.30		2 hrs.	
-10	1.3	2.7×10^{-4}	2.20	.12	2.08		3.5 hrs.	
-12	1.2	4.4×10^{-4}	2.28	.18	2.10		5.5 hrs.	
44-1	1.4	2.2×10^{-4}	2.22	.14	2.08		24 hrs.	
-8	1.6	1.4×10^{-4}	2.22	.14	2.08		31 hrs.	

TABLE 4*

AGING STUDY OF 5/D7 COATING SOLUTION MINUS D260

Exp. No. 1769-	Gamma	AEI	Dmax B	Dmin B	Dnet B	△Tr sec.	Age Days	Comme
45-1	1.4	1.1	2.30	.22	2.08	110	0	-
45-2	1.2	2.0	1.95	.24	1.71	130	.16	-
46-2	1.2	1.5	2.30	.25	2.05	120	2	-
47-1	2.0	.67	2.20	.28	1.92	120	3	-
47-4	1.4	8.5	2.60	.35	2.25	140	4	Some Blotch
48-1	1.4	1.6	2.36	.24	2.12	130	8	-
49-4	1.6	.32	1.50	.24	1.26	140	15	-
49-5	1.8	6.3	2.40	.37	2.03	130	15	Some Blotch
1824-1-2	1.5	1.2	2.31	.21	2.10	210	23	Used Another D260
5-1	1.9	.61	2.24	.20	2.04	190	30	-
9-1	1.5	.35	1.94	.24	1.70	210	37	-
13-2	2.0	.25	1.50	.16	1.34	130	44	Used Another D260
18-5	2.8	.38	2.40	.34	2.06	130	52	Some Blotch

* Reprinted from Monthly Summary Report, February, 1972.

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TABLE 5

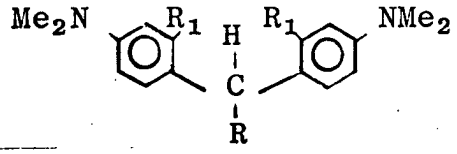
PRINTOUT EXPOSURE COMPARISON OF LMG AND ANALOGS TO LCV

No.	Dye	Amount (mg)	Dmax	Dmin	Step	Age (hrs.)	Description	Experiment Number
1	LCV	90	1.52(G)	.06	8	-	violet	1824-40-4
2		90	0.18	.06	3	3	"	-7
3		150	1.62	.06	8	-	"	-3
4		150	0.20	.06	3	3	"	-6
5	LMG (D413-C)	80	0.11(R)	.06	4	-	gray	1824-45-10
6	D413	80	0.20	.05	7	-	blue-gray	-11
7	D411	83	0.67(G)	.06	7	-	violet	1874-1-1
8	D404-C	90	0.10(R)	.06	2	-	green	1756-47-1
9	D404	90	0.12	.06	3	-	"	"
10		150	0.13	.07	4	-	gray	1824-44-2
11		150	0.07	.07	4	3	"	-5
12	D407-C	96	0.18(R)	.06	5	-	green	-45-6
13	D407	96	0.16	.05	6	-	gray	-7
14	D405-C	83	0.16(R)	.05	6	-	green	1756-47-2
15	D405	83	0.23	.05	6	-	"	"
16		150	0.37	.06	7	-	blue-gray	1824-44-3
17		150	0.10	.06	4	3	gray	-6
18	D406-C	83	0.16(R)	.06	5	-	green	1756-47-3
19	D406	83	0.21	.06	6	-	"	"
20		150	0.26	.06	6	-	blue-gray	1824-44-4
21		150	0.08	.06	3	3	gray	-7
22	D409-C	90	0.11(R)	.09	3	-	pale blue fog	1824-46-6
23	D409	90	0.06	.06	0	-	no image or fog	"
24	D408-C	110	0.15	.14	2	-	blue and fog	1824-46-5
25	D408	110	0.06	.06	0	-	no image or fog	"
26	D410-C	101	0.22(G)	.16	4	-	violet and fog	1824-45-8
27	D410	101	0.24	.14	4	-	"	-9

C = crude
B = Wratten 94 blue filter
G = Wratten 93 green filter
R = Wratten 92 red filter

TABLE 6

STRUCTURES OF LMG AND ANALOGS FOR TABLE 5



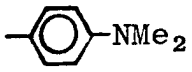
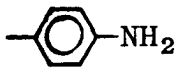
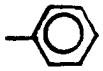
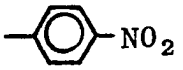
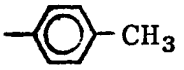
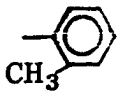
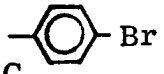
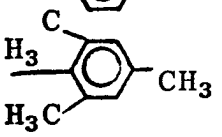
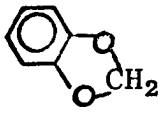
Leuco Dye	R	R_1	Molecular Weight
LCV		H	373
D411		H	345
LMG(D413)		H	330
D404		H	375
D405		H	344
D406		H	344
DD407		H	399
D409		H	372
D410		H	420
D408		CH_3	458

TABLE 7

VISCOSITY MEASUREMENTS OF PRESSURE CHEMICAL's POLYSTYRENES

Materials (10% Benzene)	Viscosity	Dry Coating Thickness (Unloaded) (Mil)
MX4500	44.0	.16
20.4K	2.4	Couldn't Be Coated
110	15.4	.19
200	23.5	.16
390	95.0	.19
498	148	.19
670	264	.19
2,000	Couldn't Be Measured	Couldn't Be Coated

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A	.06	.06	.06	.06	.08	.33	.70	1.01	1.23	1.39	1.51	1.64
B	.07	.07	.07	.18	.42	.70	.92	1.12	1.27	1.40	1.58	1.69
C	.74	.80	.82	.84	.86	.90	.94	1.04	1.11	1.20	1.33	1.43
	1	2	3	4	5	6	7	8	9	10	11	

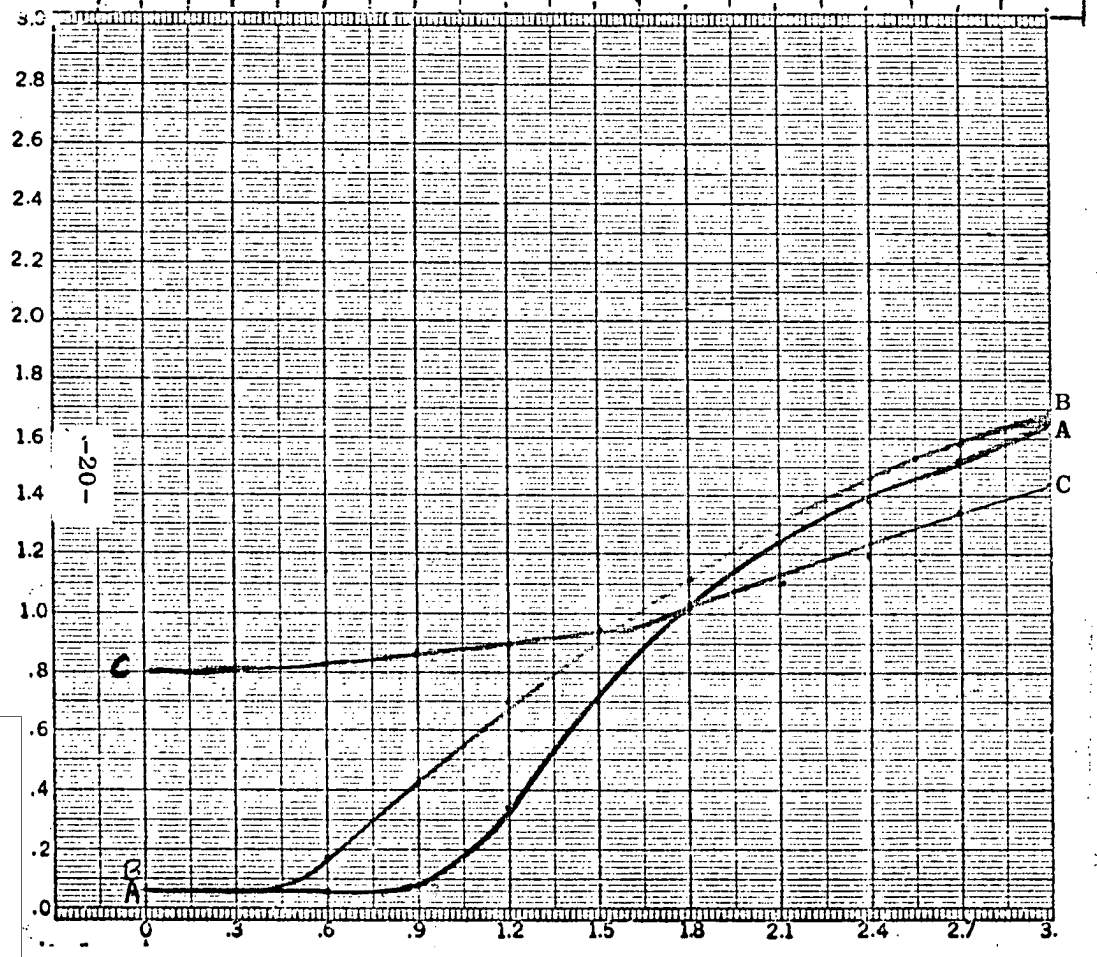

FIGURE 1 - A,B, C

FORMULA 5/DPA
PRINTOUTS

A) 1569-35-D260 (Control

B) CS-3W-D260 (ØH Rex'd)

C) 15R-D260 (Cyclohexane Rex'd)

γ = A -1.1; B - 1.1; C - .3

AEI: A - 4.2×10^{-4} B - 2.8×10^{-3}
C - 1.0×10^{-4}

.24	.24	.24	.24	.24	.24	.54	1.43	1.57	1.62	1.72	1.78	A
.10	.10	.10	.10	.10	.10	.18	.34	1.20	1.50	1.62	1.67	B
	1	2	3	4	5	6	7	8	9	10	11	

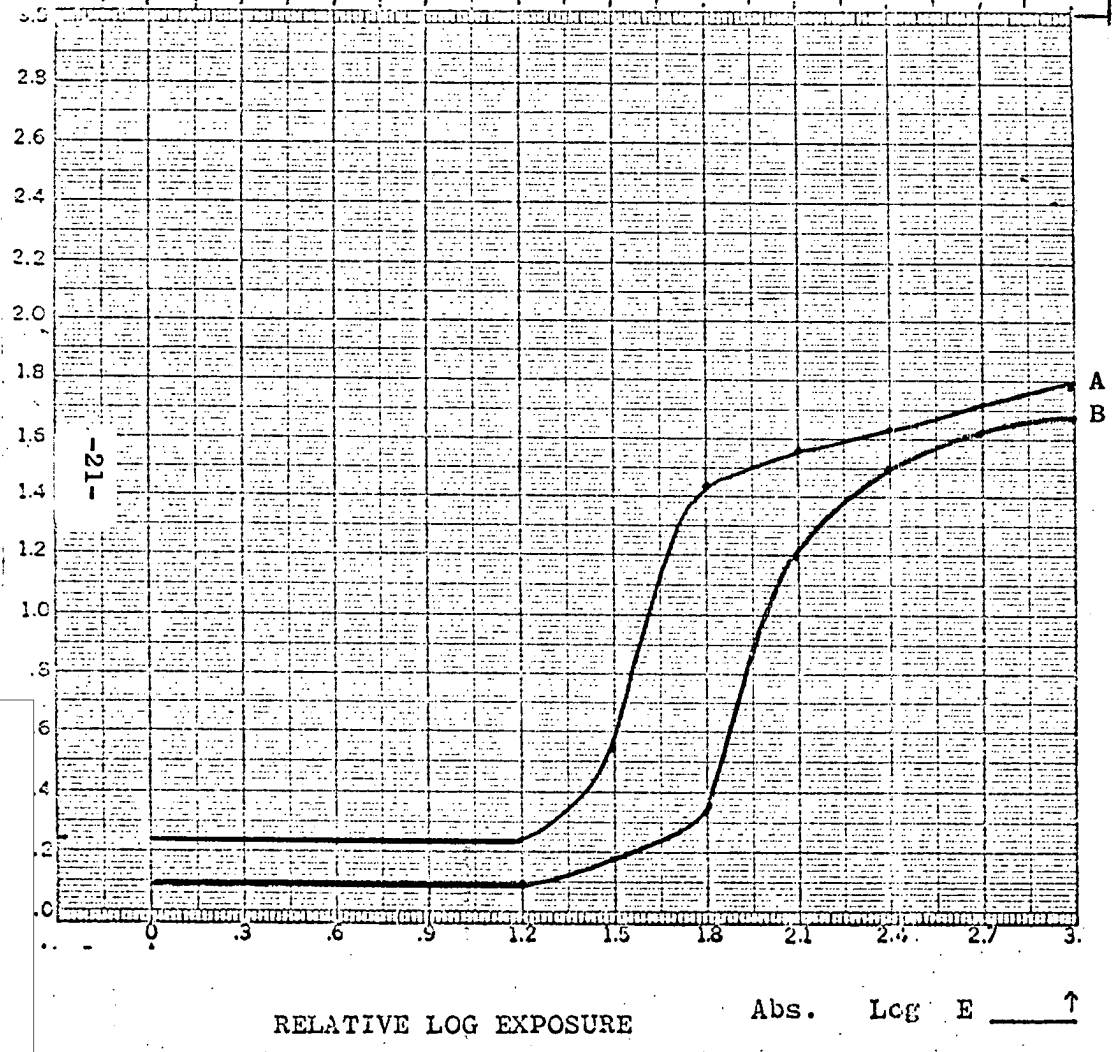

FIGURE 2 - A,B

FORMULA 5/DPA
DEVELOPMENT

A) 1569-35-D260 (Control)

B) CS-3W-D260 (ØH Rex'd)

γ = A, 3.0 B, 3.0

AEI: A, .12 B, .16

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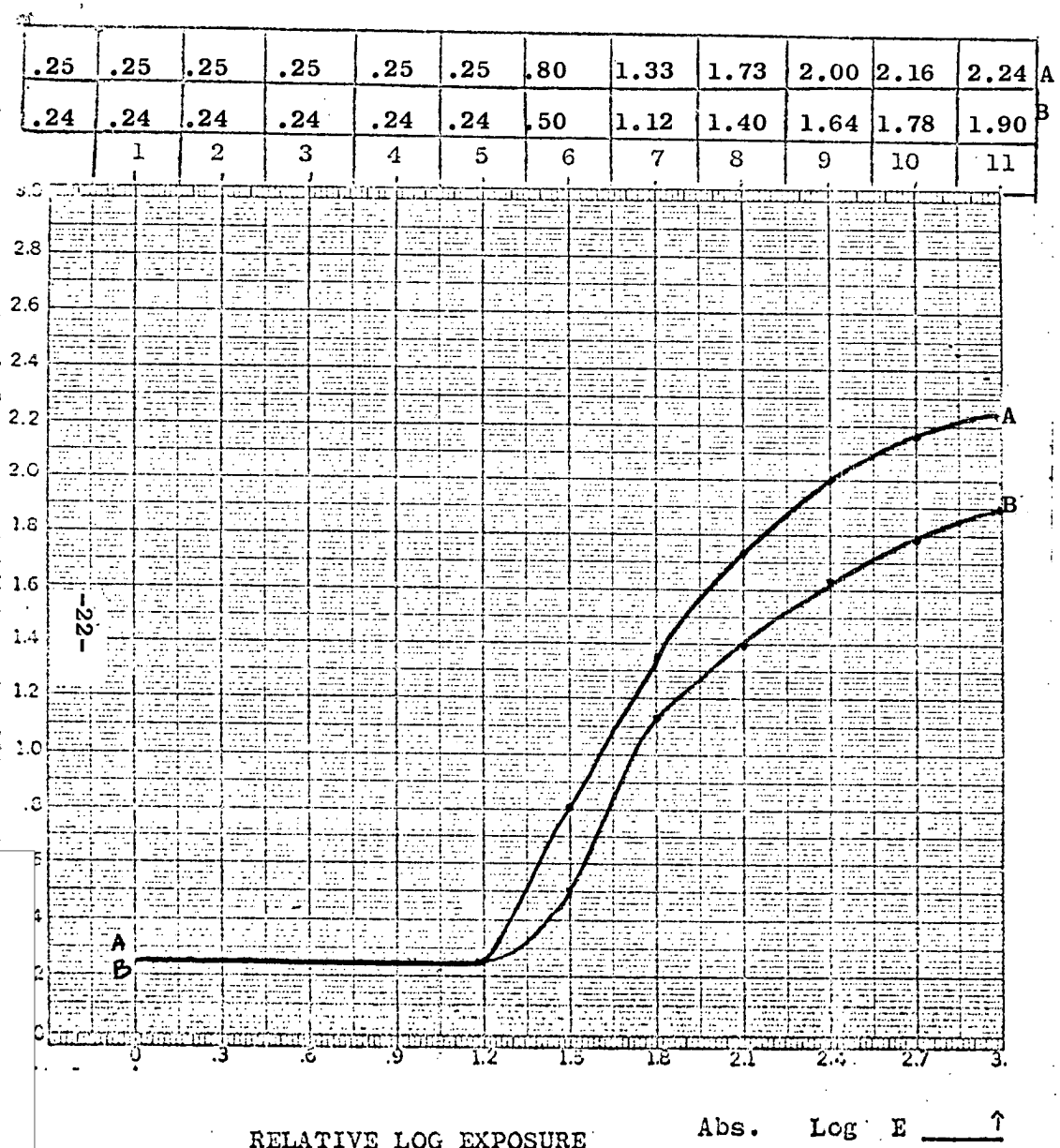

FIGURE 3 - A,B

FORMULA 5/D7
DEVELOPMENTS

A) CS-3W-D260 (ØH Rex'd)

B) 15 R-D260 (Cyclohexane Rex'd)

γ =	A, 1.8	B, 1.8
AEI:	A, 1.2	B, .92

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2.0 ENGINEERING

The major effort regarding 325B shelf life speed decay in the Engineering group has been divided into two separate areas 1) the general characteristics of the printout and red light speed decay curves for Formula 5/D7, and 2) a series of tests which might point to the reason for speed decay and/or to improve the speed decay curve.

Figure 4 shows the printout and optically developed speed decay curves for the present Formula 5/D7 system with room temperature storage. As is obvious the AEI amplification of 20,000 with fresh film has decreased to a factor of 1 after 48 hours at room temperature storage. The exact curve shape of the red lighted samples may differ slightly from the generalized curve shown. However, the beginning and end points appear valid and leave little doubt as to the bounds of the problem.

Several separate attempts have been made to improve the speed decay curve for optically developed samples. The two approaches which have shown the most promise are: 1) the storage of the samples in a high concentration of CBr_4 vapors, and 2) a one minute treatment of aged samples with benzene vapors before exposure. This has been done on both printout and red light developed samples. The optically developed samples in particular show promise. Figure 5 shows the typical speed decay curve for optically developed samples and above it the decay curve for samples stored in the can filled with CBr_4. This shows basically an improvement after 10 hours storage of a factor of 10 over untreated samples. Beyond 10 hours, few data points were taken, however, it does not appear that the CBr_4 storage will maintain the factor of 10 for periods of greater than 48 hours.

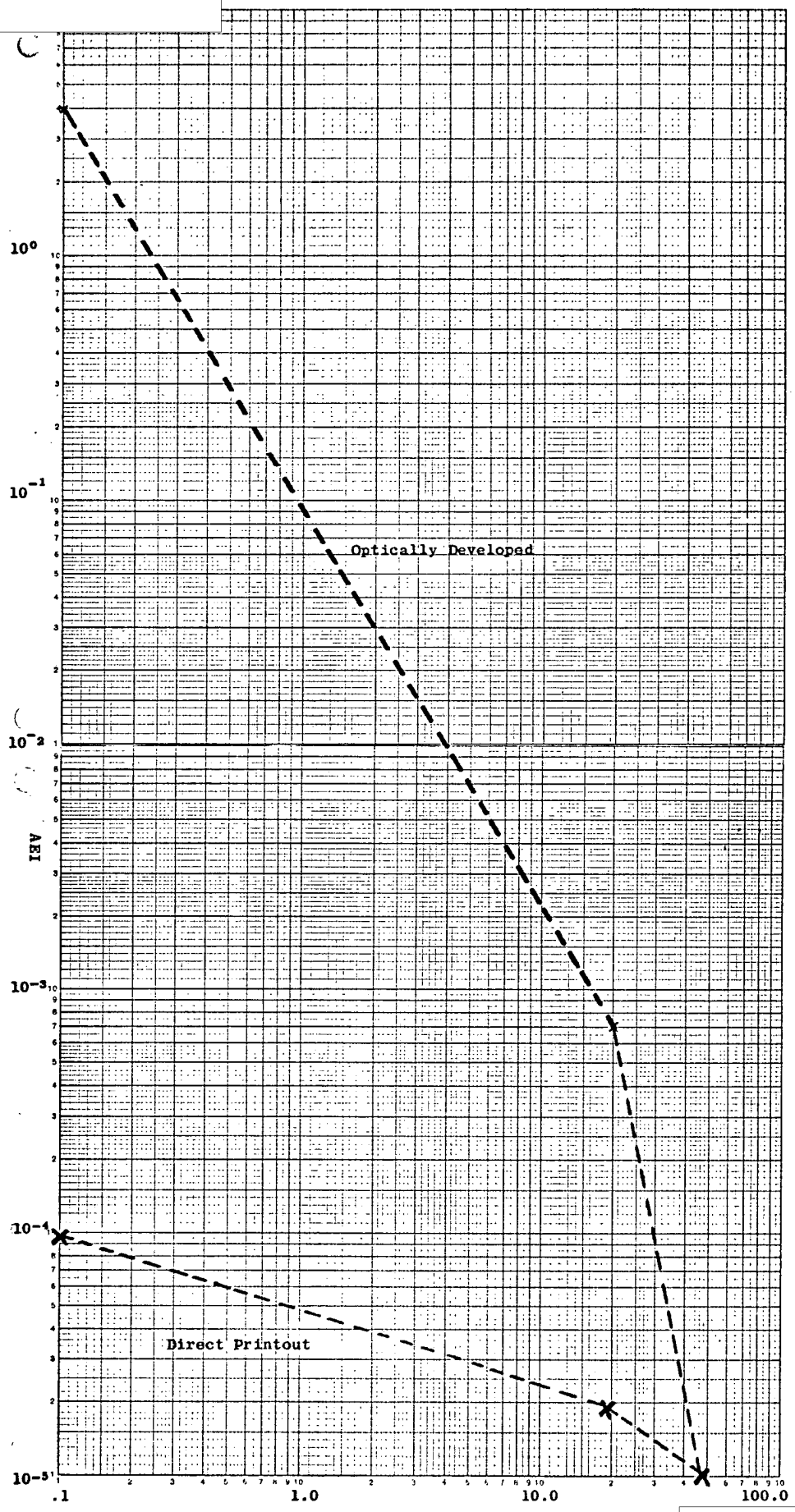
Storage of films in CBr_4 vapors is probably a technique for preventing deactivation of the film. An alternate attempt to revive the film after storage at room temperature might be called reactivation. Treatment of film samples with benzene vapors would fall in this category. Films which have been allowed to age for various periods of time were placed in a benzene vapor atmosphere for periods of up to 5 minutes, then exposed and red lighted. It was found that this technique in itself showed considerably better results than the storage of films in CBr_4 vapors. This was especially true at periods of up to 20 hours. Figure 6 shows the normal red light decay curve. The decay curve for samples stored in CBr_4 vapors and the decay curve for samples treated with benzene vapors. There is an obvious

improvement in the total system speed decay curve, however, at 20 hours there is still only an amplification of about 1,000 out of an initial 20,000. It is hoped that some combination of storage in CBr_4 vapors and then benzene treatment before exposure will show an improved speed decay curve.

Interestingly enough the benzene vapor treatment of aged samples run in the printout mode showed no improvement over the controls which were not treated.

FIGURE 4

AEI vs. TIME



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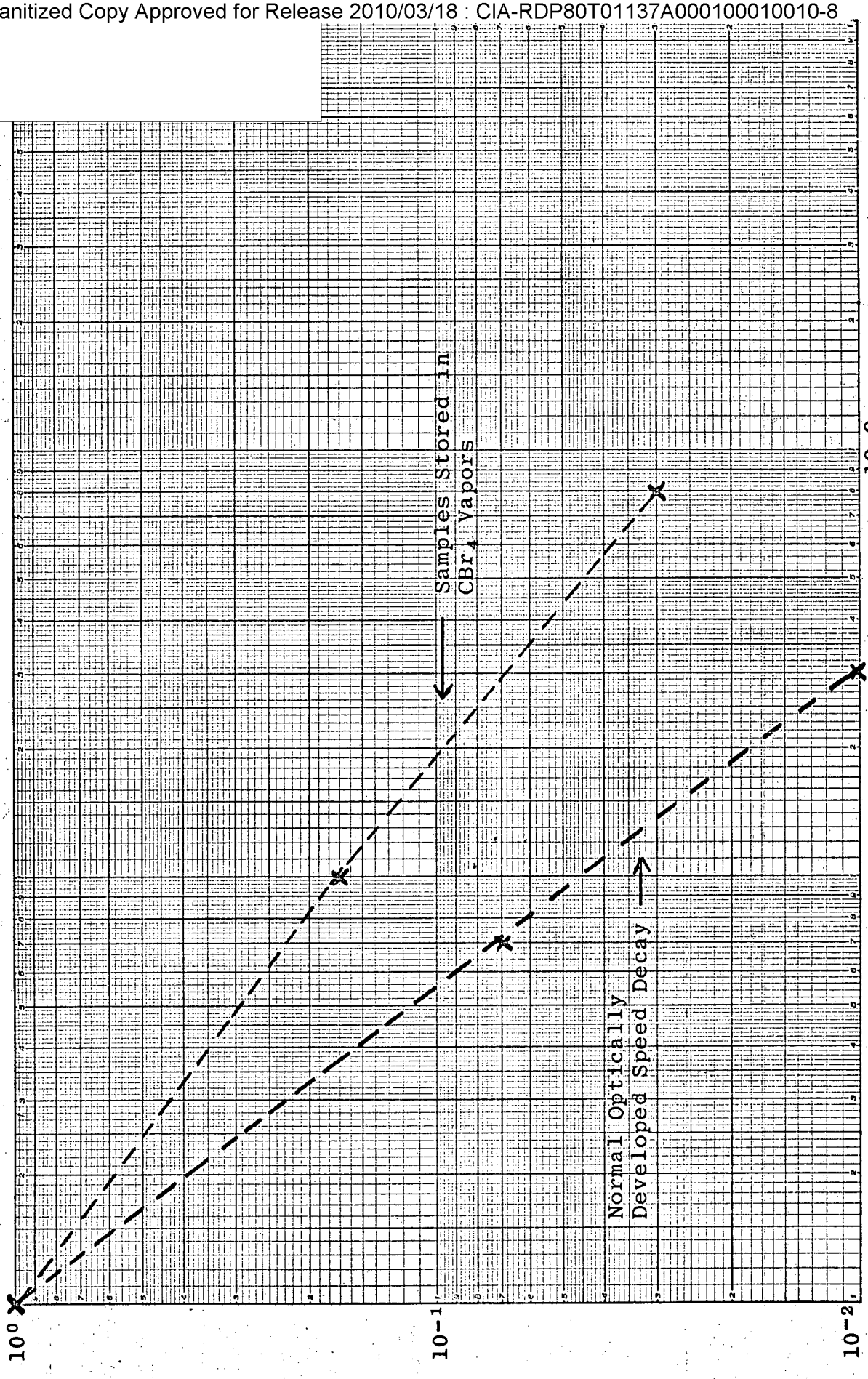

FIGURE 5

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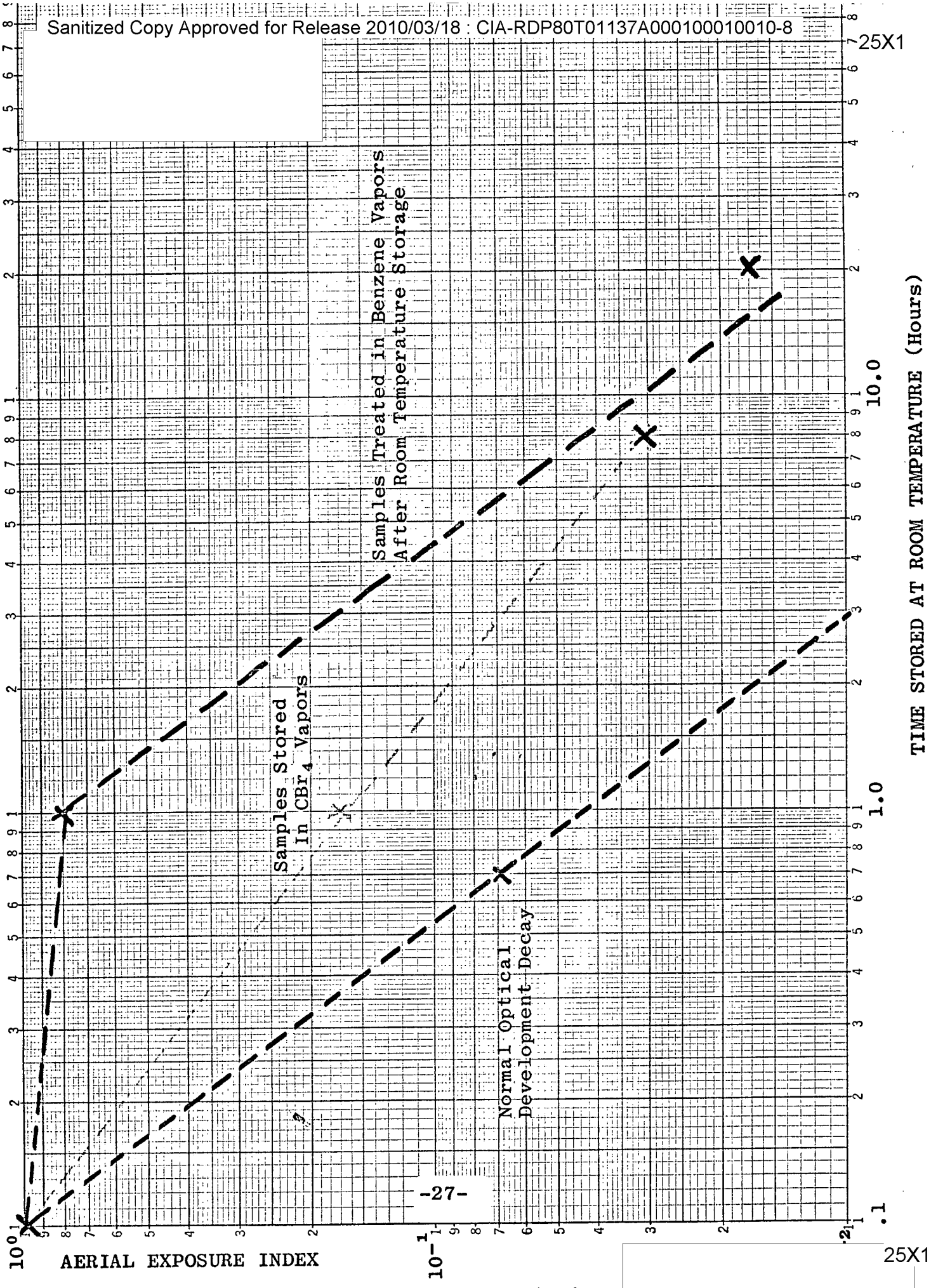
FIGURE 6



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3.0 PERKIN ELMER INTERFACE, QC/QA

3.1 Supply of Film and Equipment

3.1.1 Delivery of 325B Film

Shipment No.	Date Coated	Date Received	Time Lapse	% Fogged In Storage
1	14 Jan	17 Jan	2 Days 17 Hours	10
2	31 Jan	3 Feb	2 Days 17 Hours	100
3,4	14 Feb	16 Feb	1 Day 17 Hours	30
5	1 Mar	7 Mar	5 Days 17 Hours	0
6	15 Mar	16 Mar	17 Hours	0
7	28 Mar	29 Mar	17 Hours	0
8	17 Apr	18 Apr	17 Hours	0
9	8 May	9 May	17 Hours	

To date the originally planned number of shipments have been made (App.1, Chart 6). They have all been the same standard laboratory formulation used for R and D purposes at [redacted] 25X1

3.1.2 Delivery of Solvent Rinse

The next shipment in mid May will provide P. E. with adequate supply.

3.1.3 Delivery of Red Lite Development Units

Two additional replacement lamps were provided the 4th of May. The HID-2 unit appears to be working well and does not appear to be related to problems discussed in following sections.

3.2 Cross Calibration

Further effort to cross calibrate the [redacted] and P.E. densitometers is not within the scope of the current project. The problem has been identified and quantified. For comparisons of results between the two facilities, data will be transposed and/or reread on both densitometers. 25X1

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A new calibrated lamp has been placed in the P. E. EK-101 sensitometer. With the color correction and heat absorbing filters in place the intensity now is approximately 200 meter candles. The unit has not been measured for an absolute value with a properly calibrated photometer. The old lamp was found to be down in intensity approximately 30 to 40%. The 40% correction of the data already generated will provide a small step in bringing the P. E. and [redacted] corresponding results closer. More important will be the fact that the reduced intensity of the old lamp is an indication that the blue intensity is low. A cursory study of this effect in 1970 at [redacted] showed that the blue intensity was down eight times the amount that the photopic intensity was down when the reduction was due to aging. The effect on the film was even greater. P. E. will check out the magnitude of the problem shortly.

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3.3 Evaluation of Production, Shipping and Contamination

Production is now being performed in the engineering enclosure. Improved techniques and coordination with the format and procedures of the user have yielded smoother coatings, even though they are not filtered, and an increase from 6 to about 10 samples per sheet. When the shipment consists of a full set of 15 sheets, the 130 to 150 samples that can be extracted from the twice-a-month shipments yield as many or more tests samples as can be efficiently handled by an average coating and test technician at [redacted]

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No further problems have been experienced with shipping. A new address is now being used and the pickups are made within 17 hours of coating, although the packages do not arrive in the lab for replenishment of Dry Ice and beginning of testing until 40 hours after shipment.

Contaminants at the P. E. facility "appear" within limits or negligible. Close observations of the samples during development in the P. E. lab indicate that air contamination is probably not a problem, although the evidence is not sufficient enough to rule out the possibility (see Section 3.7).

3.4 Monitoring of Results and Anticipation of Subsequent Plans

[redacted] spent three days, April 19, 20 and 21, at the P. E. facility to monitor results and observe first hand the laboratory procedure and transient effects. [redacted] in turn visited [redacted] facility for three days, May 3, 4 and 5. It was necessary for him as a new researcher on the [redacted] film to have an appreciation of the state-of-the-art, especially

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the hand coating production technique and the different approaches used here for evaluating the film for different purposes, e.g. chemical synthesis, photographic and engineering.

The major problem that surfaced during [redacted] trip to P. E. resulted from the comparisons of the AEI speed of their red lite developed (RLD) results with the [redacted] controls and experience. P. E.'s printout results and their most recent decay curve correspond closely with [redacted] results. The primary manifestation of the discrepancy is the very low speed of their RLD samples (see Table 8). The current differences appear to be real, whereas the earlier discrepancies were masked by a considerably different densitometer, lack of color correction filters in the sensitometer, questions concerning air contamination and calibrations. The photopic intensity calibration of the P. E. EK-101 sensitometer has been indirect up to now. The meter candle value used at the time of the visit has been revised downward from 289 meter candles to approximately 120 meter candles, which still leaves a major difference in film speed results.

P. E.'s RLD tests (Table 8) show a speed that is approximately 100 to 1000 times slower than [redacted] Enough tests have been done to show a consistent discrepancy, and a rereading with [redacted] densitometer confirms this. P. E.'s RLD D-Log E curve shows a much higher γ and Dmax. Part of the discrepancy is explained by the different densitometer. As one can see. (Table 8) even with [redacted] densitometers the γ's of 6 to 7 are significantly higher than we experience here, even though γ's of 3 and 4 have been fairly common the last few months. Tied in with this is the Dmax, which shows the same type of discrepancy. The RLD curve (Figure 7) shows a shoulder at the 19th step, which is much more obvious when using P.E.'s densitometer. It happens consistently and has raised a question with P.E. Also note the dip at the 8th, 14th, and 21st step, which is due to the fact that rearranged step tablet format slightly overlaps the RLD format causing a reduction of development at those steps.

[redacted] observed a series of tests in which a matrix of exposure and development times were used. The development times of the optimum exposure was within the range experienced at [redacted] The appearance of the steps was fairly typical in that when the first step started, the following two steps appeared in sequence, i.e. not all at once (blotch). [redacted] found that the optimum RLD time varies inversely with the exposure time, when optimum RLD time is defined as base + fog of less

[redacted]

than .3 and no blotch. He has also noted that the criticality of getting the exact RLD time varies inversely with the exposure time. Exposure times were varied from 1/5 of a second, which is the basic shutter speed in the EK-101, up to printout times. Exposure times of at least 2 seconds were necessary; preferably 5 seconds, to get some developed image. Ten seconds seemed to yield optimum results (Table 8).

The shelf life study is a repeat of what [redacted] did earlier this year, except that [redacted] used the blue and heat absorbing filters in the EK-101. The printout curves and the decay curve look very much like [redacted] had three points at 0, 3 and 24 hours on a decay curve produced. They overlap very closely to [redacted] results, and the most recent results made by [redacted] Engineering group.

In order to take out a possible effect of the sequence in which the films were coated or the sequence in which the films were used for testing, [redacted] plotted the printout speeds in both orders. The speeds fell within a factor of 3, and in both cases the plots appeared fairly random indicating no effect of either sequence. In order to be consistent with regard to the amount of room temperature time, he has worked out a procedure in which he slices up his sheet into eight samples at one time. He then waits till the time lapse equals three minutes, so that each subsequent sheet is cut up in the same amount of time. They are then placed back on Dry Ice and the samples are taken out a second time, one at a time for use. The samples when removed the second time, are allowed to remain at room temperature for four minutes in order to allow sufficient drying for facedown exposure.

The identification of the speed discrepancy is a major concern because it implies a problem in cross calibrations or an as yet unknown photochemical phenomenon. The identification of the reason was sought through a duplication of [redacted] procedures on project at [redacted] To this end several coatings were made and stored on Dry Ice, as if they were being shipped to P.E.

The duplication of P.E.'s procedure at [redacted] during [redacted] visit gave a preliminary indication that RLD speeds were down a factor of 5 to 10 because of the storage and/or total of 10 minutes of room temperature aging. We, also, were able to break out an effect (perhaps an RLD speed loss of as much as a factor of 10) of "coating down" exposure on the EK-101 vs. "coating up" exposure on an [redacted] through-the-base sensitometer. More time, materials and laboratory space is needed for QC/QA to investigate these discrepancies.

3.7 QC/QA

The supply of chemicals and materials for standard Formula 5/D7 continues to keep comfortably ahead of usage rate. The supply of crude D260 from ChemSampCo has been reliable enough to prevent having to spend time to make the routine product. The quality also continues to improve. The work for identification and separation of purities continues to show good progress. A TLC test for one of the impurities has been incorporated as a QC check of materials before turning them over to the coating group. Film testing is still necessary for acceptance as photograde material. The materials as a whole still yield barely marginal quality coatings. The concentrated effort to improve quality of D260 has not been very fruitful in the last few months. As a result, attention is now being turned to an increased effort for purification of the other ingredients (see Chemistry Section).

Removal of air contamination from two other rooms in addition to the first chemistry room has succeeded, but evidence indicates that improvements and consistency are needed. We now have five (5) Barnebey-Cheney cabinet filtration units. The latest one to arrive, which is double in size, had to be moved into a small room in which a smaller cabinet unit was barely maintaining good levels. The engineering enclosure and the two chemistry coating rooms now are maintained at a sufficiently good level. Two of the five cabinet units help to maintain a low level of contamination in the general chemistry area by their placement in the interlock which exists before the laboratories and the large room that contains the glove boxes and environmental chamber. Chemistry units are now in operation in the D260 purification laboratory. In addition small canister units are used in the engineering enclosure interlock and the chemical purification lab. The purification lab should be sealed off, conditioned, and filtered similar to the coating labs in which the purified materials are used. The cabinet filtration units are monitored and controlled on a systematic basis (see Figure 8). About 300 lbs. of activated charcoal is used per month, since the filters are now changed monthly.

A more cost effective filtration method probably exists, but the criticality of maintaining the film research has not afforded the luxury of experimenting with the existing rooms. In the latter half of 1971 the charcoal filtration units have picked up a considerable amount of chlorides and bromides, as well as Tellurium, oxidants, Sulfur dioxide and Nitrogen dioxide (see Figure 9). That, plus the evidence illustrated in the frequency diagrams of the air sampling, plus the evidence of acid contamination

of stock solutions, strongly indicates that acid gases are the air contaminant.

Cause of the high γ experienced at P. E.'s facility might tie in with three other conditions in which we experience high γ as a positive test for "bad" film. Bad D260, contaminated laboratory air, and aged films in [redacted]'s lab all show an increased γ as at least one of their defects. This grouping interprets the "blotch" as an extreme case of high γ. Although each of the four conditions that yielded high γ's can be differentiated by close observations the suggestion being made is a common cause. The differences then would be looked upon as subvarieties or extraneous effects.

The intensive work with purification and identification of purities in D260 suggest the possibility that an impurity might be the cause in all four cases, whether autogenerated or absorbed. In particular, the chemical purification group has reported increased amounts of a particular impurity upon storage of D260 in a stock solution. An example, strictly for illustrative purposes at this point, of a common cause could be an acidic condition due to aging of the materials, and/or absorption from the atmosphere which in turn could be cause or effect in the chemistry of a bad coating. "Impure" D260 usually results in decreased Dmax along with higher γ in the RLD mode. D260 serves as the example here because the other ingredients have not been investigated as thoroughly yet.

"Bad" films due to air contamination usually show "blotching" as well as high γ. The high γ's associated with increased exposure have been observed with film that has lost a considerable part of its original printout sensitivity. The shipped films used by P. E. have retained the printout speed but lost a considerable part of the RLD speed in addition to having a much higher γ. If high γ represents an explosive type quantum yield as illustrated in this section of last month's report, then a chemistry mechanism study might also find conditions that accelerate the production of dye. A relatively simple test that might add some validity to the theory of common cause is a TLC check for one or two of the specific impurities already identified. This test, which is well documented for the purification of materials and has also been done for aged solutions, will be done for aged solids and also of film produced in "clean" vs. "dirty" rooms and short vs. long age times on or off ice.

TABLE 8

COMPARISON OF [redacted] CONTROLS and P. E.'s RESULTS

Shipment Number	Exposure Sec.	Exposure M. C.	RLD Sec.	AEI	γ	Dmax	Densitometer TD-102
Red Lite Developed:							
7	10	120**	45	1.1×10^{-3}	14.3	2	Perkin Elmer
7	10	120	60	2.2×10^{-3}	9.5	2	Perkin Elmer
7	10	120	75	8.7×10^{-3}	26.4	2	Perkin Elmer
8*	10	120	45	2.3×10^{-3}	6.8	2.4	[redacted]
*8, Control	.088	220	40	0.65	2.0	1.95	
7, Control	.09	188	49	1.4	3.8	2.5	
Printout:							
7, Control	300	188	-	2.2×10^{-4}	1.8	2.5	[redacted]
7	180	120	-	2.4×10^{-4}			Perkin Elmer
*8, Control	300	220	-	1.8×10^{-4}	1.8	2.4	[redacted]
8, Control	300	220	-	0.9×10^{-4}	2.1	2.6	
*8	180	120	-	2.6×10^{-4}	1.3	2	

Controls were exposed and processed at [redacted] All others were exposed and processed at Perkin Elmer.

* These are plotted in Figure 7 .

** 120 m. c. is the value currently estimated to have been correct. 289 m. c. was the value used at the time of the April visit.

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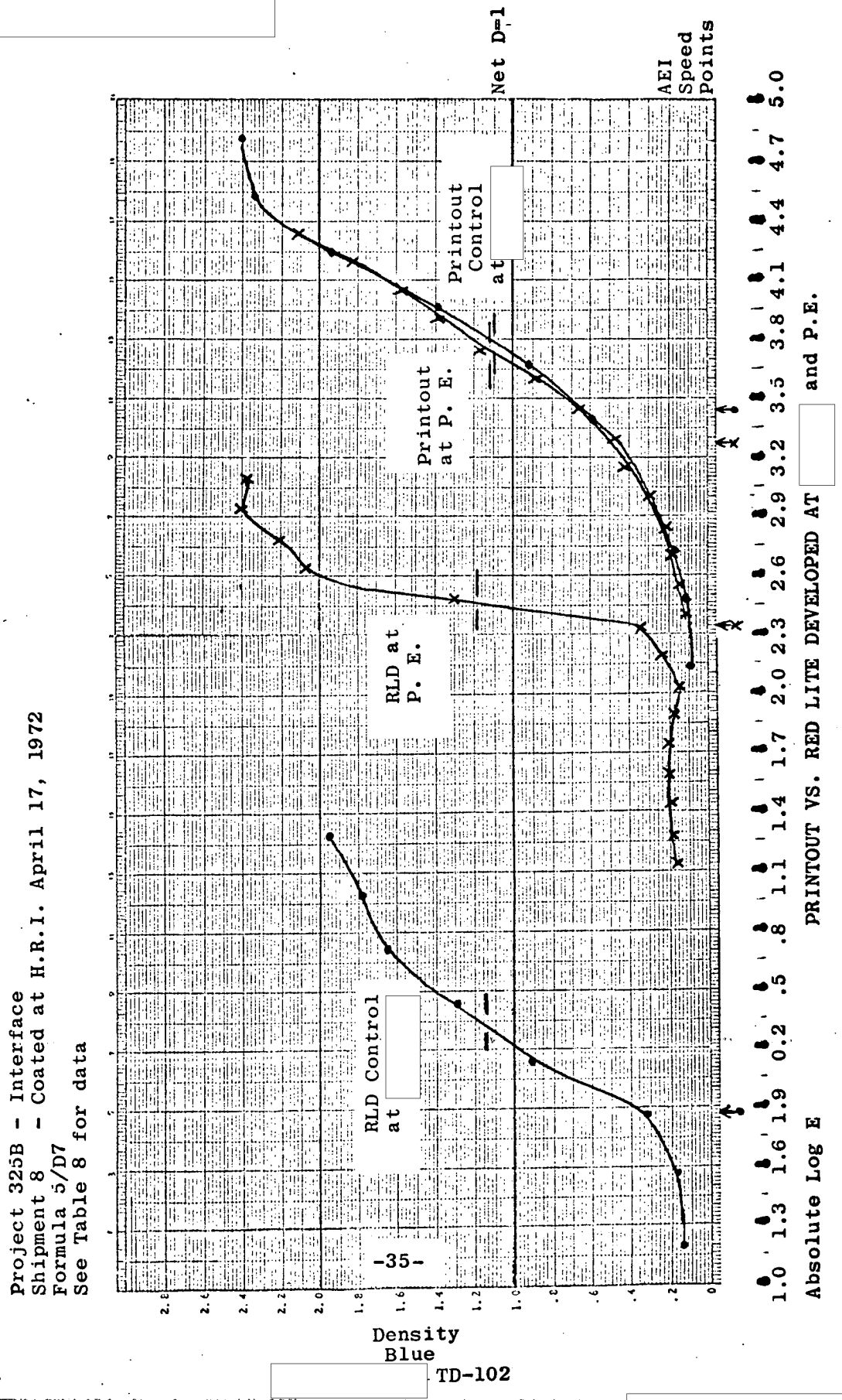
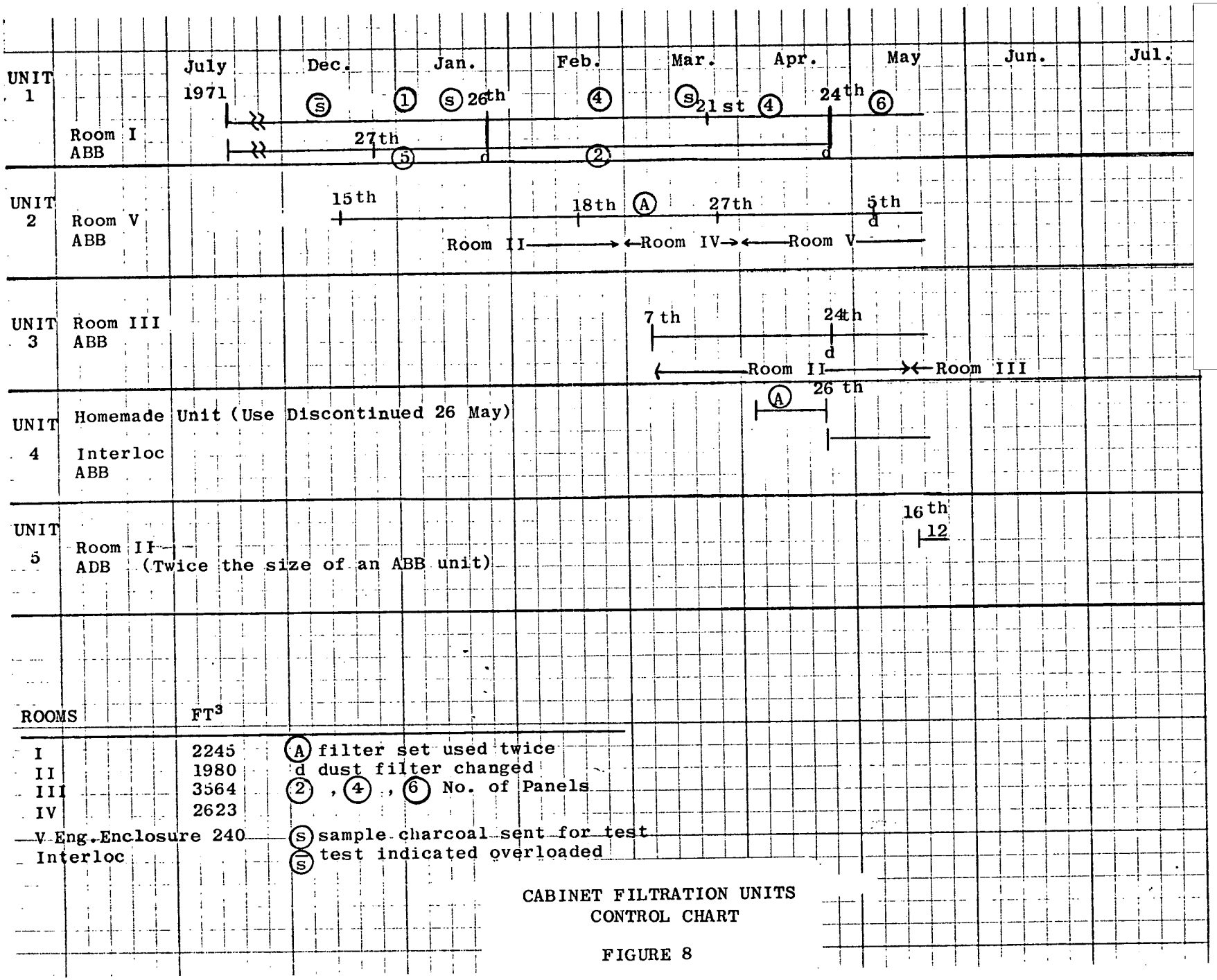


FIGURE 7

Project 325B - Interface
Shipment 8 - Coated at H.R.I. April 17, 1972
Formula 5/D7
See Table 8 for data

TD-102



CABINET FILTRATION UNITS
CONTROL CHART

FIGURE 8

FIGURE 9

CONTAMINANTS PICKED UP BY ROOM AIR FILTRATION UNIT

Contaminant	Amt./Kg of Charcoal
Chloride ion	90 grams
Bromide ion	21.7 grams
Tellurium	14.1 grams
Oxidants as O_3	42 milligrams
Sulfur dioxide	13 milliliters
Nitrogen dioxide	2.4 milliliters

Tests for Copper, Zinc, Iron, Manganese, Nickel, Chromium, Cobalt, Silver, Lead, Cadimium, and Arsenic were negative.

A six lb. sample of activated charcoal was taken from the 42 lbs. when the first unit's filters were changed the first time after 5-1/2 months of operation in the small chemistry coating lab (Room I), and analyzed along with a sample of fresh unused activated charcoal by National Loss Control Service Corporation. The filtration unit is a Barnebey Cheney Cabinet Purifier Model No. ABB which was run at 400 cfm. and contains 42 lbs. of charcoal.

PROBLEMS

None.

PLANS FOR NEXT REPORTING PERIOD

Continue project work along forecast schedule.

FINANCIAL

Project is tracking within labor and materials budgets. See project tracking graphs.

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[redacted]

FINANCIAL STATUS SUMMARY

PROJECT 325B

MAY 1, 1972

25X1

[redacted]

Note: Material was committed under the 325A contract for delivery in 1972 and for use on the follow-on project 325B. After the fact, these charges were not accepted under 325A, and, as they had not been planned for in the current program, they have now been added to the May forecast. Thanks to a small underrun in direct Labor, it is not expected that this will increase the final cost of the program when overhead and G and A are calculated.

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[redacted]

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APPENDIX 1

1) Legend for Gant Charts 1 through 6

2) Examples for Gant Charts 1 through 6

3) 1.0 Chemical R and D: Charts 1 through 5

4) 2.0 Engineering: Chart 5

5) 3.0 Perkin Elmer Interface, QC/QA: Chart 6

LEGEND FOR GANT CHARTS 1 THROUGH 6

HEAVY LINE: Major Section and Major Subsections

THIN LINE: Subsections (Tasks)

Solid Line: Completed to date

Dashed Line: Remainder of forecast period plus extension(s)

Wavy Line: No work performed

1) though originally forecast
2) subsequent to existing date indicates will be abandoned without immediate plans for rescheduling

▽ Forecast completion date

▼ Completed on schedule

△ Extended completion date

▲ Completed extension

Task performed but at lower level of effort than forecasted

Extended into second six months

1,2 ect. - Explanatory note (see bottom of page)

x(y) x = Forecast manpower loading
y = Actual manpower loading
Major Sections only

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EXAMPLES FOR GANT CHARTS 1 THROUGH 6

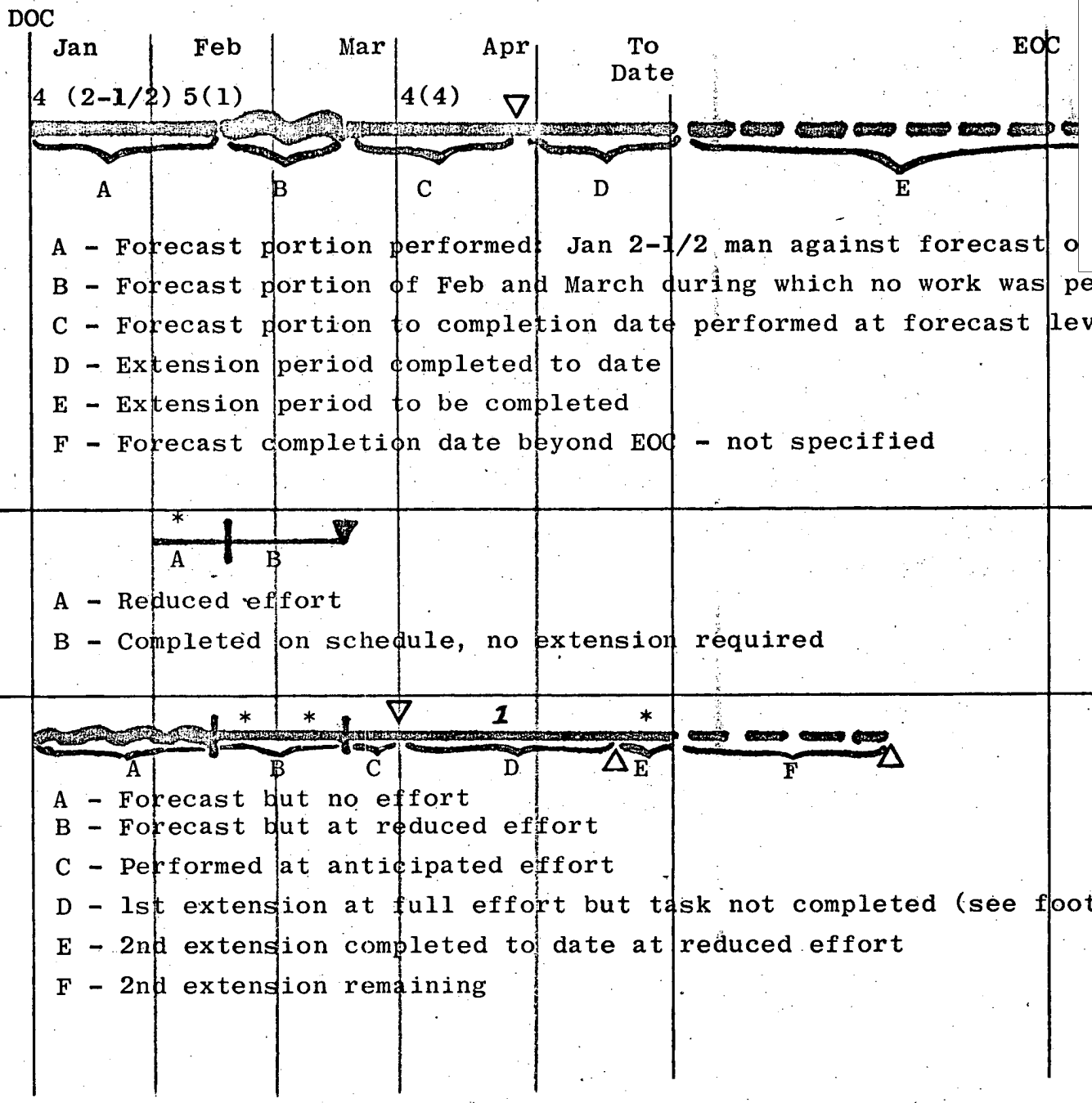
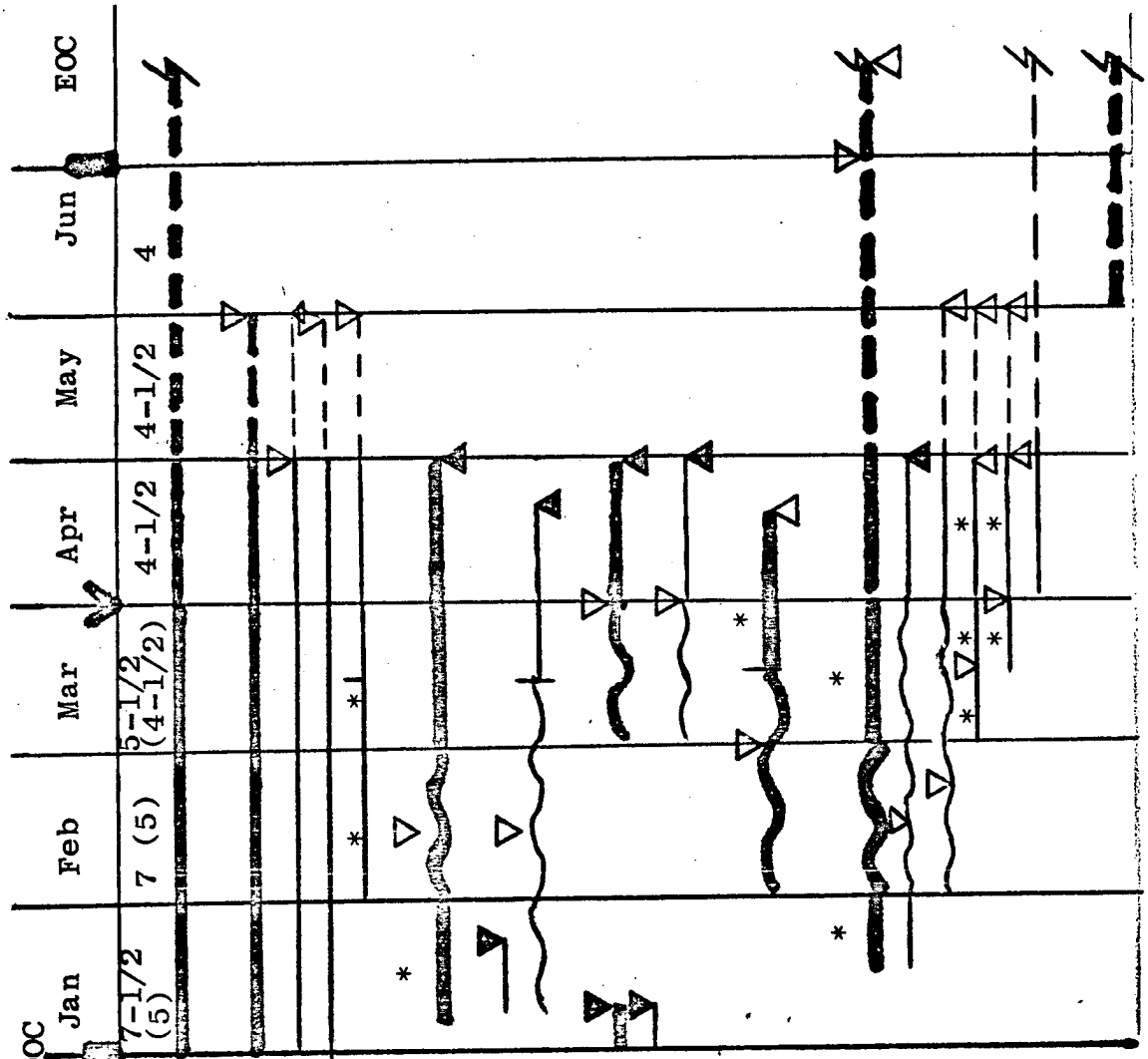


CHART 1



1.0 CHEMICAL R AND D

1.1 Leuco Dye Program

1.1.1 D260
1.1.1.1 Synthesis and Purification
1.1.1.2 Impurity Studies
1.1.1.3 Alternate Synthesis

1.1.2 Leuco Malachite Green (LMG)
1.1.2.1 Synthesis
1.1.2.2 Purification

1.1.3 LMG Congeners
1.1.3.1 Syntheses
1.1.3.2 Purifications

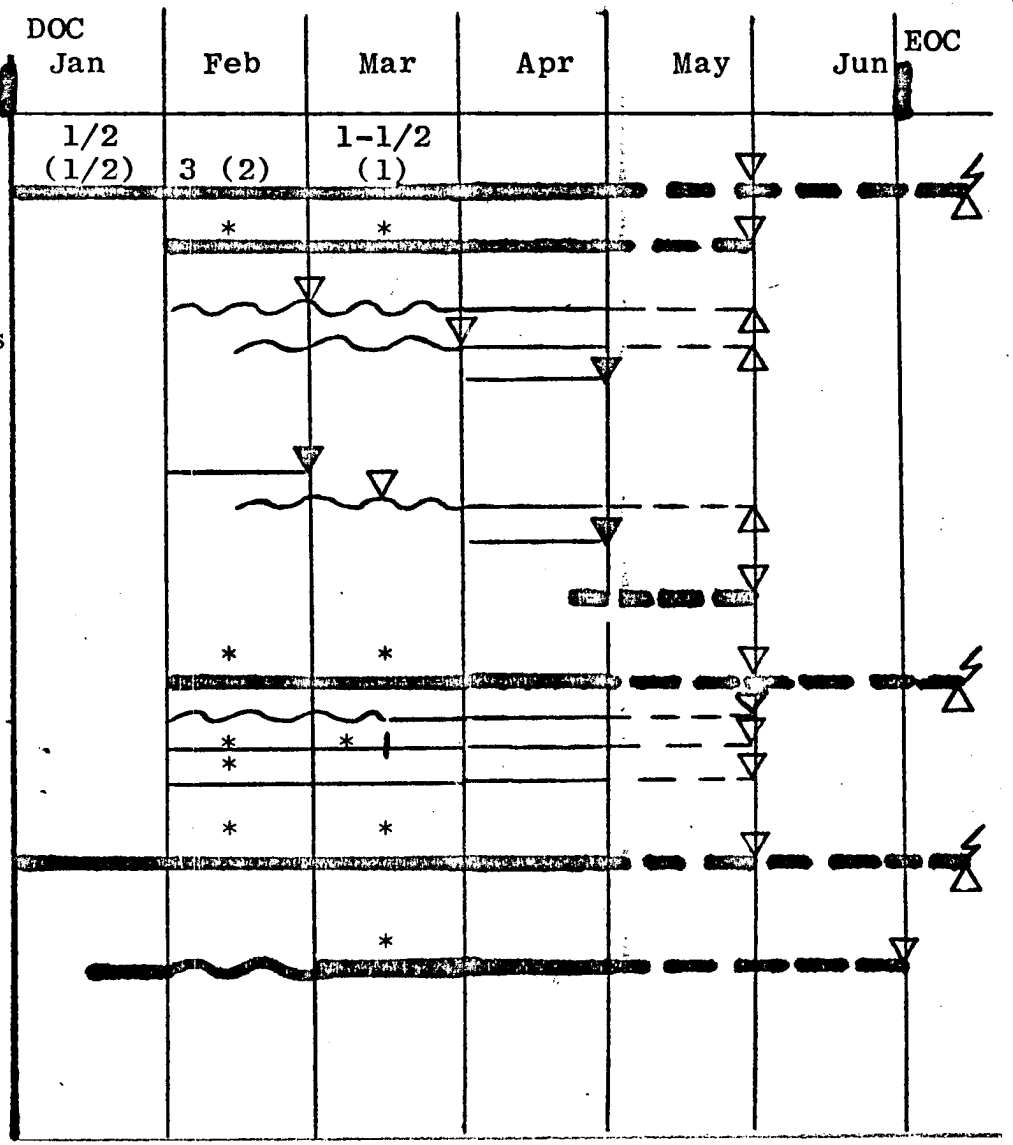
1.1.4 Leuco Crystal Violet (LCV)

1.1.5 LCV Congeners
1.1.5.1 D259
1.1.5.2 D174
1.1.5.3 2-Methyl
1.1.5.4 2,6-Dimethyl
1.1.5.5 Others

1.1.6 D260 Congeners

CHART 2

CHEMICAL R AND D (continued)



1.2	CBr_4 Activator Program
1.2.1	Purification Studies
1.2.1.1	(Freeman Crude)
1.2.1.1.1	Purified Atmosphere
1.2.1.1.2	Rex'n from Other Solvents
1.2.1.1.3	Oxygen-free Atmosphere
1.2.1.2	(BDH)
1.2.1.2.1	Purified Atmosphere
1.2.1.2.2	Rex'n from Solvents
1.2.1.2.3	Oxygen-free Atmosphere
1.2.1.3	(Others)
1.2.2	Deterioration Studies
1.2.2.1	High Vacuum Storage
1.2.2.2	Pure Atmos. Storage
1.2.2.3	Normal Storage
1.2.3	Detection and Identification of Impurities
.2.4	Other Activators

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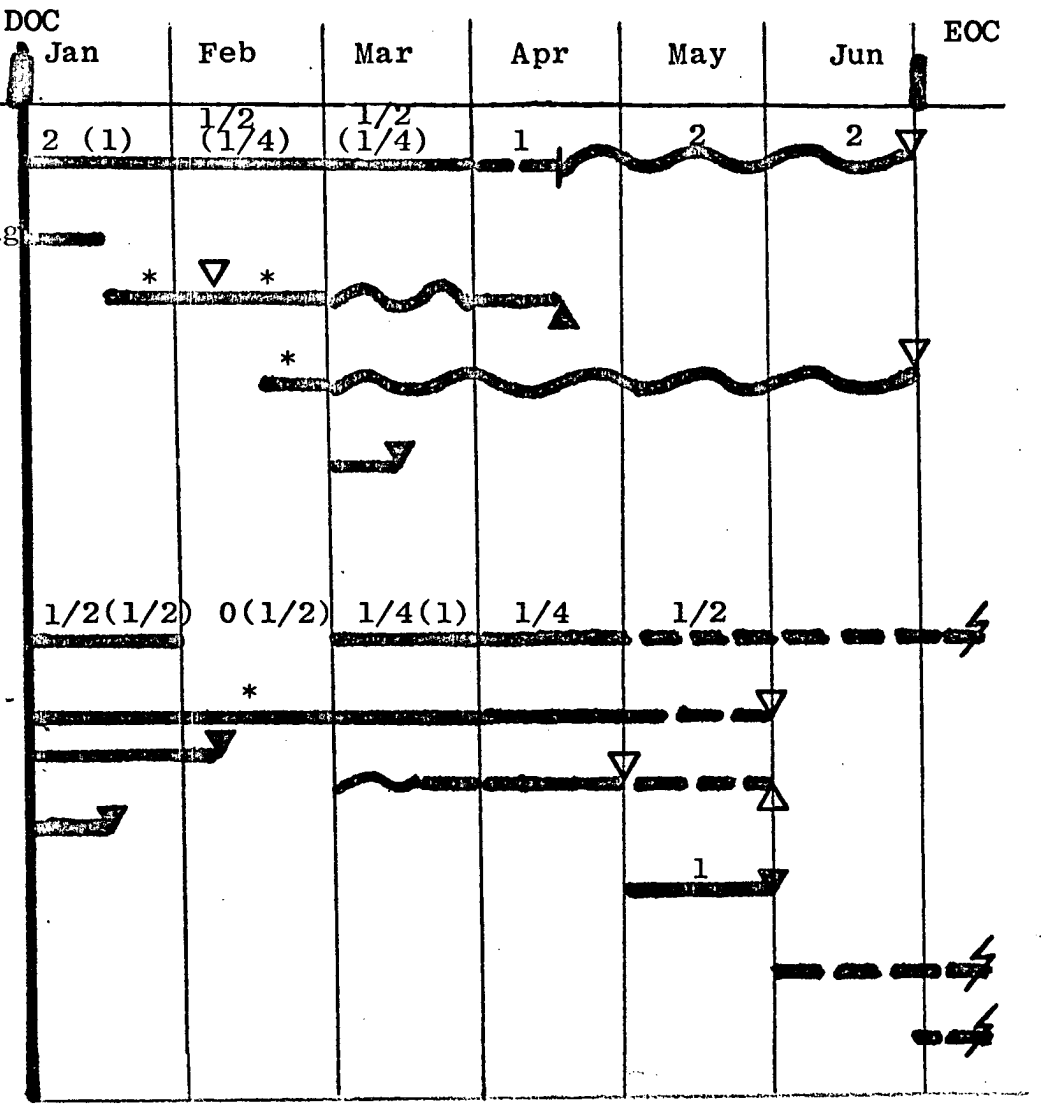
CHART 3



1 - Completed ahead of Schedule: In February.

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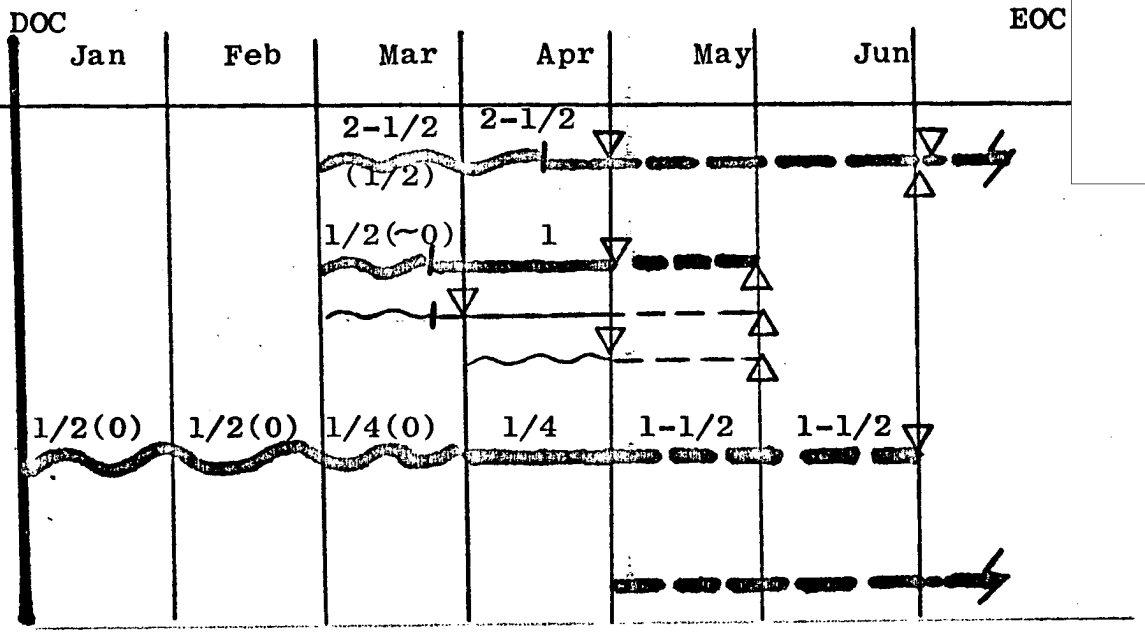
CHART 4

CHEMICAL R AND D (continued)



1.5 Environmental Studies

1.6 Other Film Systems

1.6.1 5/DPA

1.6.2 7/D7, 5/D8

1.7 Film Analyses of Decay Products

1.8 Dye Identification

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CHART 5

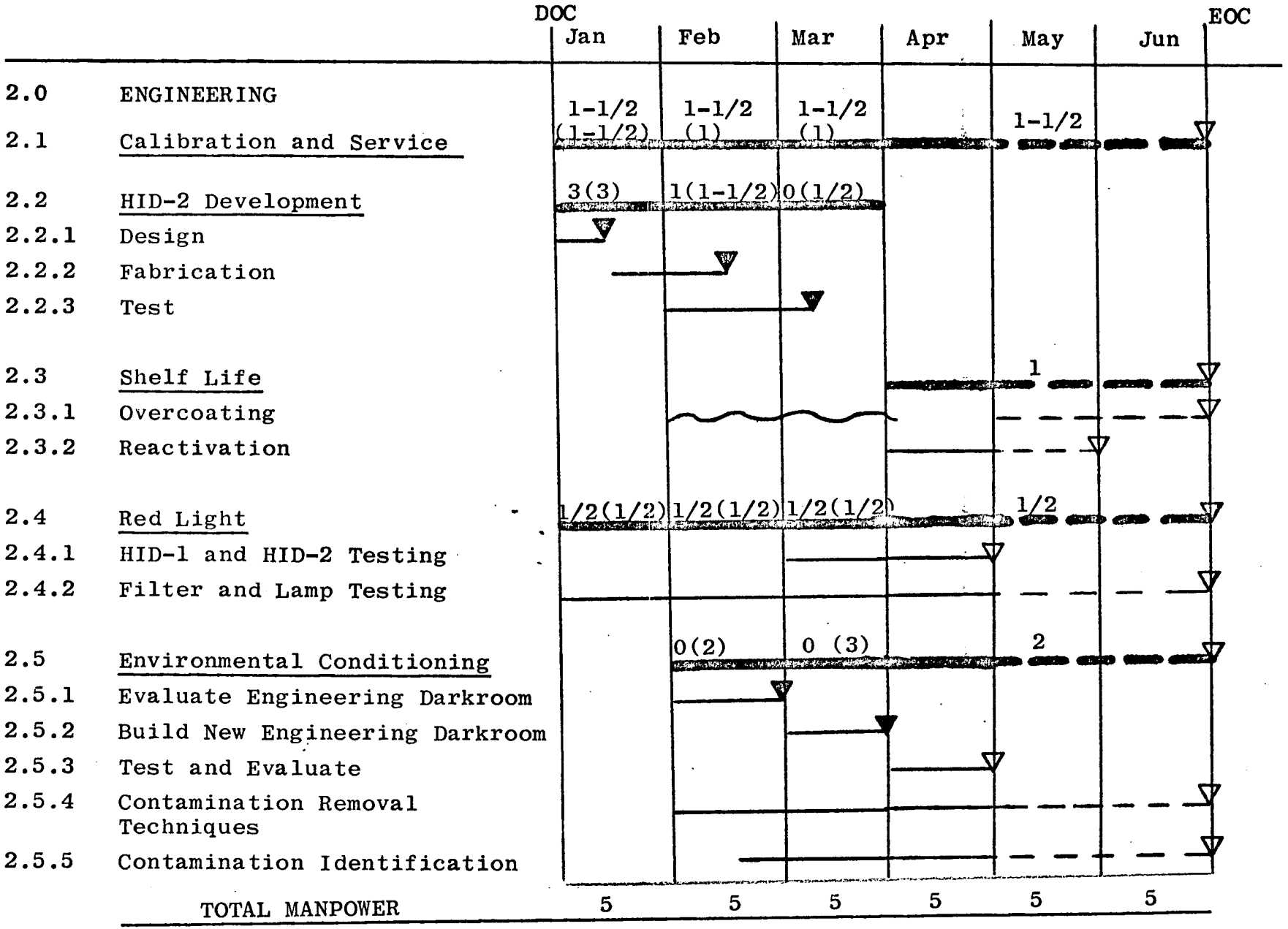

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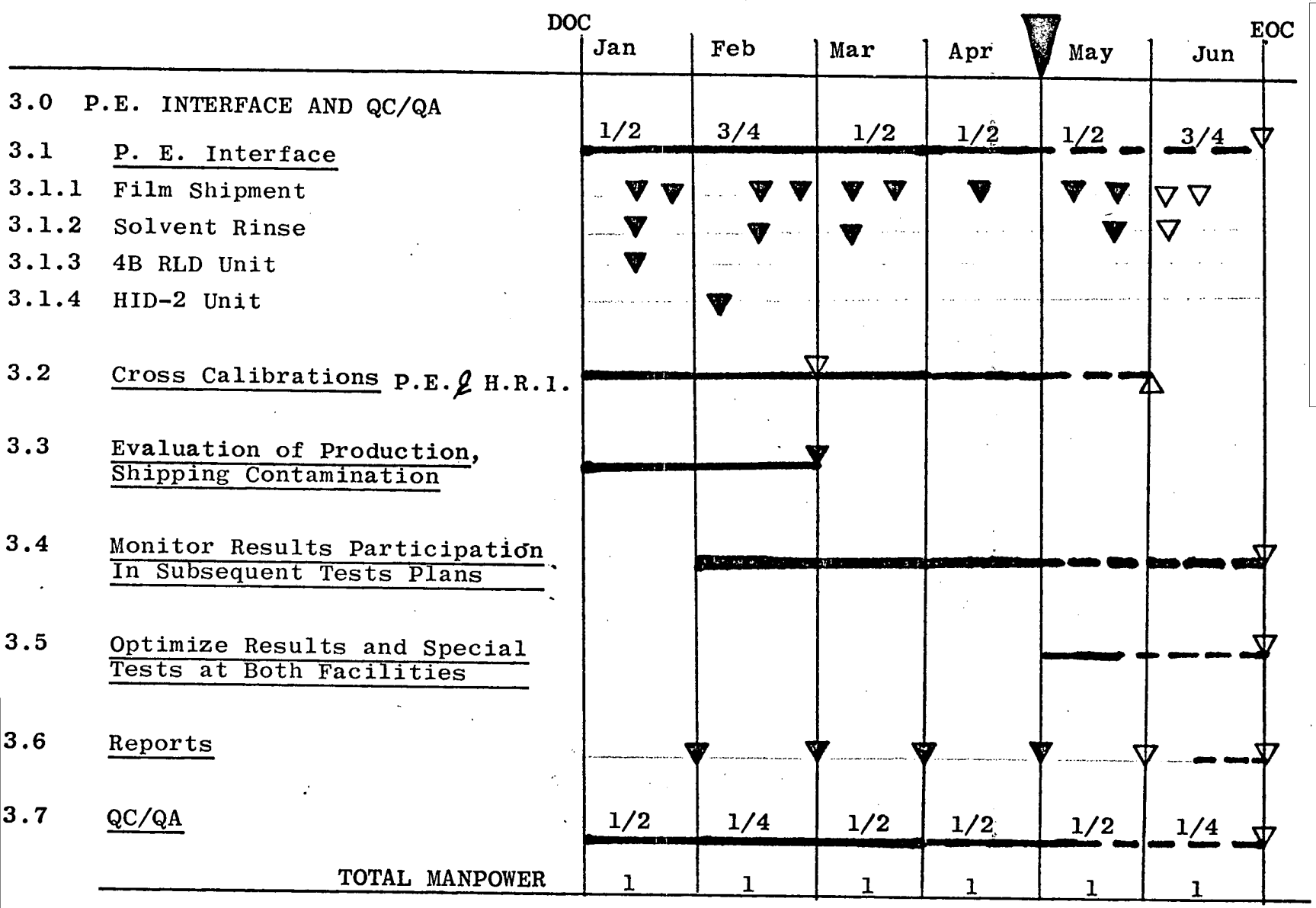
CHART 6



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